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In re application of

**ANDREW JOHN HOLMES and
CAMERON WILLIAM WATSON**

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HYDRAULIC FLUID

Group Art Unit: 1764

Examiner: Ellen M. McAvoy

Attorney Docket: TS7564

COMMISSIONER FOR PATENTS

P. O. Box 1450

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Sir:

**DECLARATION OF RICHARD DIXON UNDER
37 C.F.R. § 1.132 IN RESPONSE TO OFFICE ACTION
MAILED NOVEMBER 15, 2005**

I, Richard Dixon, declare as follows:

1. I am a research scientist employed by Shell International Petroleum Company Limited in Chester, United Kingdom.
2. I received a Ph.D. degree in Chemistry from the University of Edinburgh in 1997. My doctoral research work focused on catalysis of ethylene. My thesis dissertation was entitled "Reflection Absorption Infrared Spectroscopy Investigations on the Ni(111) surface." All of the research was in the field of petrochemicals, sponsored by a petrochemicals company.
3. I have been employed as a research chemist with Shell Research Limited ("Shell") in the lubricants and hydraulic fluids area since 1996. I am employed as a research scientist and Technology Manager in its Lubricants division. In this capacity, I am involved in the development of new products such as lubricants and hydraulic fluids.
4. I am an author of publications, in the lubricants and hydraulic fluids area, some of the publications are as follows:

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SAE 2002-01-1456, *Developments in Shear Stable Hydraulic Fluids*, R. T. Dixon and K. J. Young, Shell Global Solutions, presented in Las Vegas, USA.

SAE 2000-01-2651 *Environmental Standards for Biodegradable Hydraulic Fluids and Correlation of Laboratory and Field Performance*, N. Battersby, R. T. Dixon, S. Greenall, C. W. Watson and K. J. Young, I. Elm, T. Marougy, presented in Milwaukee, USA.

5. Based on my education and experience, I am qualified to express any opinions set out below.

EXTENDED WEAR TESTING

6. Shell conducts an annual or bi-annual investigation of fluids available in local and global markets. Samples of fluids are obtained from the market and tested in a range of common tests, normally including elemental and infrared analysis to determine the fluid composition. The tests are performed at Shell laboratories in Cheshire (United Kingdom) and Atsugi (Japan).

7. During the course of a Shell annual or biannual investigation, extended wear testing was conducted in order to understand the long-term performance of hydraulic fluids. The tests were performed over a duration of 1000 hours instead of 250 hours according to the procedures set forth in IP 281/80, "Determination of anti-wear properties of hydraulic fluids - Vane pump method," (Exhibit A).

8. I supervised the testing referred to in the foregoing paragraph at the Shell laboratory in Cheshire. I am in frequent communication and make physical visits to the laboratory in Japan. I am satisfied that the extended wear testing adhered to high scientific standards.

9. During the extended wear testing, the "Test Sample" had the following composition:

0.012 %wt magnesium salicylate having a total base number of 345 and a magnesium content of 550% of the stoichiometrically equivalent amount of magnesium based on the amount of total acid, containing 40 wt.% mineral oil;

0.25 wt.% zinc dithiophosphate ("ZnDTP"), and

99.05 wt % lubricant API Group I base oil having a kinematic viscosity in the range of 32 cSt (at 40 °C) (ISO viscosity grade 32).

10. The "Test Sample" was compared to competitive products comprising no identifiable metal salicylates by the analysis performed and varying amounts of zinc (as ZnDTP).

11. The results of the tests are shown in the Chart entitled Extended Anti-Wear Test, attached as Exhibit B. The amount of zinc in the competitive products is indicated in the legend.

12. As seen from Exhibit B, only the Test Sample (comprising ZnDTP and magnesium salicylate) exhibited consistent low total wear of below 50 mg over the entire test period of 1000 hours.

13. In contrast, total wear using commercial products comprising ZnDTP without magnesium salicylate increased over time to from about 80 to over 250 mg over the same 1000 hours. In some cases, the increase began immediately. In others, the increase began at about 500 hours.

ADDITIONAL COMPARATIVE DATA

14. Additional testing was performed using the test procedures in the Examples of the pending application:

COMPOSITION 1, a combination of ZnDTP and magnesium salicylate;
COMPOSITION 2, a combination of ZnDTP and calcium salicylate;
COMPOSITION 3, to compare the effect of magnesium salicylate alone; and,
COMPOSITION 4, to compare effect of ZnDTP alone.

The compositions tested and the results are given in Exhibit C.

15. Compositions 1 and 2 were originally submitted as comparative testing in the pending application. The testing of Compositions 3 and 4 were provided as additional testing where the total treat rate of the listed additives remained constant (0.53%) of which the antirust component remained constant at 0.10%.

16. The results obtained using Composition 3 illustrate the effect of a relatively high concentration of magnesium salicylate, alone, on wear testing under the conditions used in the Examples. Composition 3 produced total wear of 1.9 mg.

indicating that the use of magnesium salicylate (alone) in the hydraulic fluid had a significant influence on low-load performance.

17. The results obtained using Composition 4 illustrate the effect of a relatively high concentration of ZnDTP, alone, in the wear testing under the conditions used in the Examples. Composition 4 produced total wear of 8.6 mg, indicating that the use of ZnDTP (alone) in the hydraulic fluid also lowered total wear.

18. A comparison of the results obtained using Composition 3 to the results obtained using Composition 4 demonstrate that a quantity of magnesium salicylate (alone) was more effective than the same quantity of ZnDTP (alone) in reducing total wear under the conditions used in the Examples.

19. Exhibit B also illustrates that total wear using commercial products comprising ZnDTP (alone) without salicylate increased over time, as compared to the Test Sample which comprised a combination of ZnDTP and magnesium salicylate.

I understand that willful false statements and the like are punishable by fine or imprisonment, or both (18 U.S.C. 1001) and may jeopardize the validity of the application of any patent issuing thereon.

SIGNED this 15th day of May, 2006.


Richard Dixon

IP 281/80
(1988)

BS 2000 : Part 281 : 1993

Determination of anti-wear properties of hydraulic fluids – Vane pump method

This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations.

1. SCOPE

1.1 This method describes the determination of the steel-on-steel anti-wear properties of hydraulic fluids by means of a vane-type hydraulic pump. It covers a range of fluids intended for use in applications with high-speed sliding contacts such as are encountered in a vane pump.

2. SUMMARY OF METHOD

The pump is run for 250 h at one of the following two conditions depending on the fluid being tested:

2.1. A pressure of 140 bar and a fluid level of 25 to 700 mm above the centre line of the pump inlet for mineral oils (types HM and HV – see Annex XI) and synthetic fire resistant fluids (type HFD) and at a temperature to give an oil viscosity of 13 cSt at the pump inlet.

2.2. A pressure of 105 bar and a fluid level of 500 to 700 mm above the centre line of the pump inlet for other types of fire resistant fluids (types HFA, HFB and HFC). The higher pressure of 140 bar may be employed if the fluids have adequate anti-wear properties. The temperature at which the test is carried out is that required to obtain an oil viscosity at the pump inlet of:

2.2.1. 13 cSt for fluid types HFA and HFC for fluids less than a viscosity grade of 32 cSt at 40°C.

2.2.2. 30 cSt for fluid types HFA, HFB and HFC for fluids of viscosity grades between 32 and 68 cSt at 40°C inclusive.

2.2.3. 60 cSt for fluid types HFB and HFC for fluids above a viscosity grade of 68 cSt at 40°C.

This method is not intended for use with fluids that do not meet the above viscosity requirements.

The maximum temperature for conducting the test in accordance with 2.1 is recommended as 100°C. For fluids detailed in 2.2 a maximum temperature of 60°C is recommended. Special precautions regarding materials may be necessary at temperatures above 90°C, e.g. seals.

Fluid performance is assessed by measuring the weight loss of ferrous working parts (ring and vanes).

3. APPARATUS

See Annex X2 and Fig. XI and manufacturers' instructions.

3.1. *Vickers (Sperry-Rand) vane-pump* – Type 104C or 105C.

3.2. *Power input facility* – 11 kW at 1440 rpm (approx).

3.3. *Pressure loading device* – if the viscosity loss of the fluid is being evaluated during the test, the

pressure loading device should be of the needle valve type with a seat diameter of 5 mm (sharp edged) and capable of controlling the pressure between 7 and 140 bar at the operating temperature. The direction of flow through the needle valve is required to be from the small diameter end to the large diameter end of the needle.

3.4. *Pressure relief valve* – (over-load protection) – suitable for pressures up to 160 bar.

3.5. *Reservoir* – 55 to 70 litres capacity with sealed lid fitted with a filtered breather. If the viscosity loss of the fluid is being evaluated the quantity of fluid is to be 70 litres.

3.6. *Heat exchanger* – for fluid cooling capable of maintaining the temperature at the pump inlet to $\pm 2^\circ\text{C}$.

3.7. *Filter unit* – absolute rating of the element to be lower than 30 micrometres and suitable for the fluid under test.

3.8. *Flow meter* – up to 45 litres per min approximate capacity.

3.9. *Pressure measuring device* – up to 200 bar.

3.10. *Temperature measuring device* – up to 150°C.

3.11. *Analytical balance* – capable of measuring to within 0.0001 g.

3.12. *Surface plate*.

4. MATERIALS

4.1. *Solvent* – white spirit conforming to BS 245 or Petroleum Spirit 60/80 conforming to current IP specification when testing mineral oils and HFD fluids. For HFA, HFB and HFC fluids use butylglycol or isopropyl alcohol.

4.2. *Fine-grade abrasive stone* – for removing sharp edges of the pump components.

4.3. *Emery polishing paper* – Grade 2/0.

5. PREPARATION OF APPARATUS

5.1. Drain off any fluid remaining from a previous test ensuring that all parts are drained, as far as possible, and discard the filter element. If however, the previous test fluid was a different type, all the components affected are to be carefully cleaned with an appropriate cleaning fluid. Change the seals if not compatible with fluids to be used. Mineral oils and HFD fluids should be removed by petroleum spirit or white spirit. For HFA, HFB and HFC fluids use butylglycol or isopropyl alcohol.

5.2. Charge the tank with 20 litres (minimum) of test fluid.

5.3. Select a suitable used cartridge for flushing purposes, dip in the test fluid and fit to the pump.

281.1

Exhibit A

ANTI-WEAR PROPERTIES, IP 281

5.4. Fit the cover plate to the pump and tighten the bolts in increments of approximately 1 N m in the sequence 1, 5, 3, 7, 2, 6, 4, 8 until the pump binds. Slacken the bolts and re-tighten in increments of approximately 1 N m to 1 N m less than the binding torque. Check that the pump rotates freely. If it does not rotate freely slacken the bolts and re-tighten carefully.

5.5. Start the pump and run for 15 min with the pressure loading device at the lowest setting. Repeat twice (5.2) with fresh test fluid if previous test fluid was of a different fluid type.

5.6. During the flushing procedure increase the pressure in the system and adjust the relief valve (overload protection) to 10% above the intended test pressure.

5.7. Drain off the fluid as in 5.1 and fill the tank with 55 to 70 litres of test fluid as appropriate to the system to obtain fluid levels (refer Fig. X1). Fit a new filter element.

5.8. Select a new cartridge Pt No. 912014 or Pt No. 429126.¹

5.9. If cartridge Pt No. 912014 or 429126 is employed, check and record dimensions as follows:

- ring width, in four places equally spaced,
- rotor width between each vane slot,
- vane width.

The ring width should be 0.018 to 0.036 mm in excess of the rotor width, and the rotor should be 0.005 mm to 0.015 mm in excess of the vane width.

5.10. Lightly stone, to remove sharp edges:

- the vane slots in the rotor,
- the edges of the vane which run against the port plates, and
- the inner edges of the ring.

Lightly rub the faces of the port plates in a figure-eight motion on the emery paper, which should be placed on the surface plate. Reject any port plates that do not polish evenly over the outer 6 mm annulus of the surface.

5.11. Thoroughly clean the cartridge in appropriate cleaning fluid, preferably by ultrasonic means, and dry with a lint-free cloth or dry air.

5.12. Weigh all the vanes together and weigh the ring. Record the weights.

5.13. Re-assemble the test cartridge, ensuring that the chamfered edges of the vanes trail in the normal direction of rotation. Moisten lightly with the test fluid during assembly. With water containing fire-resistant fluids immerse the cartridge for 18 h in the test fluid before assembly.

5.14. Remove the cartridge used for flushing and fit the test cartridge.

5.15. Fit the pump end cover and tighten the end bolts in accordance with the sequence described in 5.4.

6. TEST PROCEDURE

6.1. Set the pressure loading device (3.3) to its lowest setting.

¹Cartridges with the number 711628 are regarded as obsolete and must not be used.

6.2. Start the pump and run the pump for one hour, at a pressure setting of 20 bar for HFA, HFB, HFC and HFD fluids and at a pressure setting of 35 bar for mineral oils.

6.3. Test Conditions and Duration.

6.3.1. Delivery pressure 140 bar or 105 bar according to the test fluid, refer section 2.

6.3.2. Temperature at pump inlet: adjust to give appropriate viscosity for fluid under test, refer section 2.

6.3.3. Pump delivery at start of test 24 to 30 litres per min.

6.3.4. Duration: 250 h.

6.4. Increase the pressure in increments of 20 bar at 10-min intervals up to the test pressure ± 2 bar. The 20 bar increments should be achieved over a period of 60 sec during each 10-min interval. Adjust the oil cooler to maintain the temperature.

6.5. If, on attaining the test pressure and temperature, the flow is below 24 litres per min, the pressure should be reduced to a minimum and the pump stopped, and re-adjusted in accordance with 5.4. This re-adjustment should be completed within five min of stopping the pump. Re-start pump and increase pressure as in 6.4.

6.6. During the test, check and record operating conditions daily. Adjust operating conditions as required.

6.7. If the flow falls by more than 25% of its original value the test should be stopped. If the stoppage is due to port plate failure the test is null and void.

6.8. On completion of the 250 h, or after earlier stoppage, remove the cartridge and wash the parts in the appropriate cleaning fluid, ensuring that any hard deposits are also removed. Dry with a lint-free cloth or dry air. Ultrasonic cleaning methods may be used.

6.9. Weigh all the vanes together and then the ring. Record the masses.

6.10. Determine the kinematic viscosity after the test in a manner to enable comparison with that at the start of the test.

6.11. Examine the parts for physical appearance.

7. REPORT

7.1. Report the mass loss of the vanes in mg and the mass loss of the ring in mg at the end of the 250 h, or after an earlier stoppage.

7.2. Report the appearance of the components.

7.3. In cases of earlier stoppage, report actual duration.

7.4. Report viscosity at two temperatures before and after the test for HV, HFA, HFB, HFC and HFD fluids. This requirement is optional.

7.5. Report: Conditions used (2.1. or 2.2.)

Pressure
Fluid level
Temperature
Cartridge number

8. PRECISION

The precision of this method has not been established.

ANTI-WEAR PROPERTIES, IP 281

ANNEX X1

DESCRIPTION OF FLUID TYPES

- HM- Mineral oils possessing particular anti-oxidation, anti-corrosion and anti-wear properties.
- HV- Fluids of form HM possessing improved viscosity/temperature properties.
- HFA- Fire resistant fluids of the oil-in-water emulsion type.
- HFB- Fire resistant fluids of the water-in-oil emulsion type.
- HFC- Fire resistant fluids of the water-polymer solution type.
- HFD- Fire resistant fluids of the synthetic type containing no water.

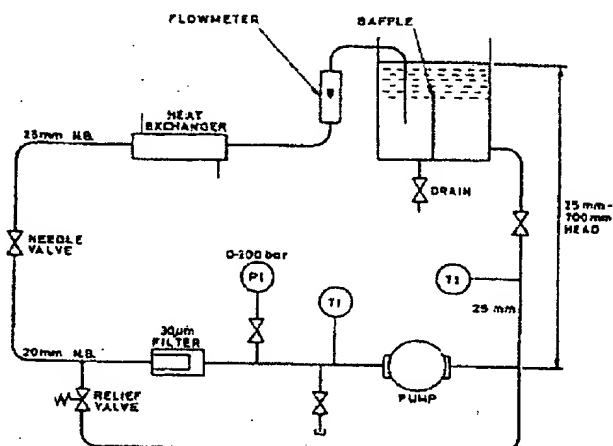


Fig. X1. Test rig.

ANNEX X2

APPARATUS

X2.1. Vickers (Sperry-Rand) Type V104-C-10/V105-C-10 Vane Pump.

X2.1.1. Test cartridges are obtainable from:

Sperry Vickers,
Frolingstrasse 41,
D6380 Bad Homburg vdH, West Germany

Sperry Vickers
PO Box 4
New Lane
Havant
Hants PO9 SNB, England

Vickers Sperry Rand Corporation
Administrative & Engineering Center
PO Box 302
Troy
Michigan 48084, USA

The cartridge is identified by the number 429126 and it should be indicated that the parts are for fluid testing and must be equipped with bronze bushing. In the USA a separate rotor is available under the part number 429446.

X2.1.2. Assembly of pump—see manufacturers' instructions.

X2.2. Vickers (Sperry-Rand) Type V104-C-10/V105-C-10 Vane Pump.

X2.2.1. Cartridges are obtainable from:

Vickers Sperry Rand Corporation,
Administrative & Engineering Center,
P.O. Box 302,
TROY,
Michigan 48084, U.S.A.

The cartridge is identified by the part No. 912014, and it should be indicated that the parts are for fluid testing, and must be equipped with bronze bushings.

GUIDANCE INFORMATION

(1) It is recommended that protection circuits are incorporated to cater for temperature, pressure and fluid level variations.

(2) A suitable needle valve for the viscosity loss evaluation is available from Alenco Industrial Components Limited, Belmont Road, Maidenhead, Berkshire, SL9 9JP, designated Ermeto Fine Control Valve Type 3, Part No. W25746 [Note 1]. The hole through which the needle passes (approximately $\frac{1}{2}$ in diameter) is required to be increased to 5 mm.

NOTE 1: This valve will require adaptors to suit the size of piping used.

(3) Equipment should be suitable for the range of fluids and temperatures to be tested, e.g. it is recommended that a stainless steel reservoir is employed and particular attention is paid to filters and seals when used at elevated temperatures.

(4) When low fluid heads are employed, it is recommended that the pump inlet pipe enters the reservoir through the side and the pipe end faces the bottom of the reservoir (to avoid vortex). For high fluid heads, it is recommended that the inlet pipe is taken from the bottom of the reservoir.

(5) A vacuum gauge is recommended near to the pump inlet.

(6) Automatic recording equipment is of considerable assistance.

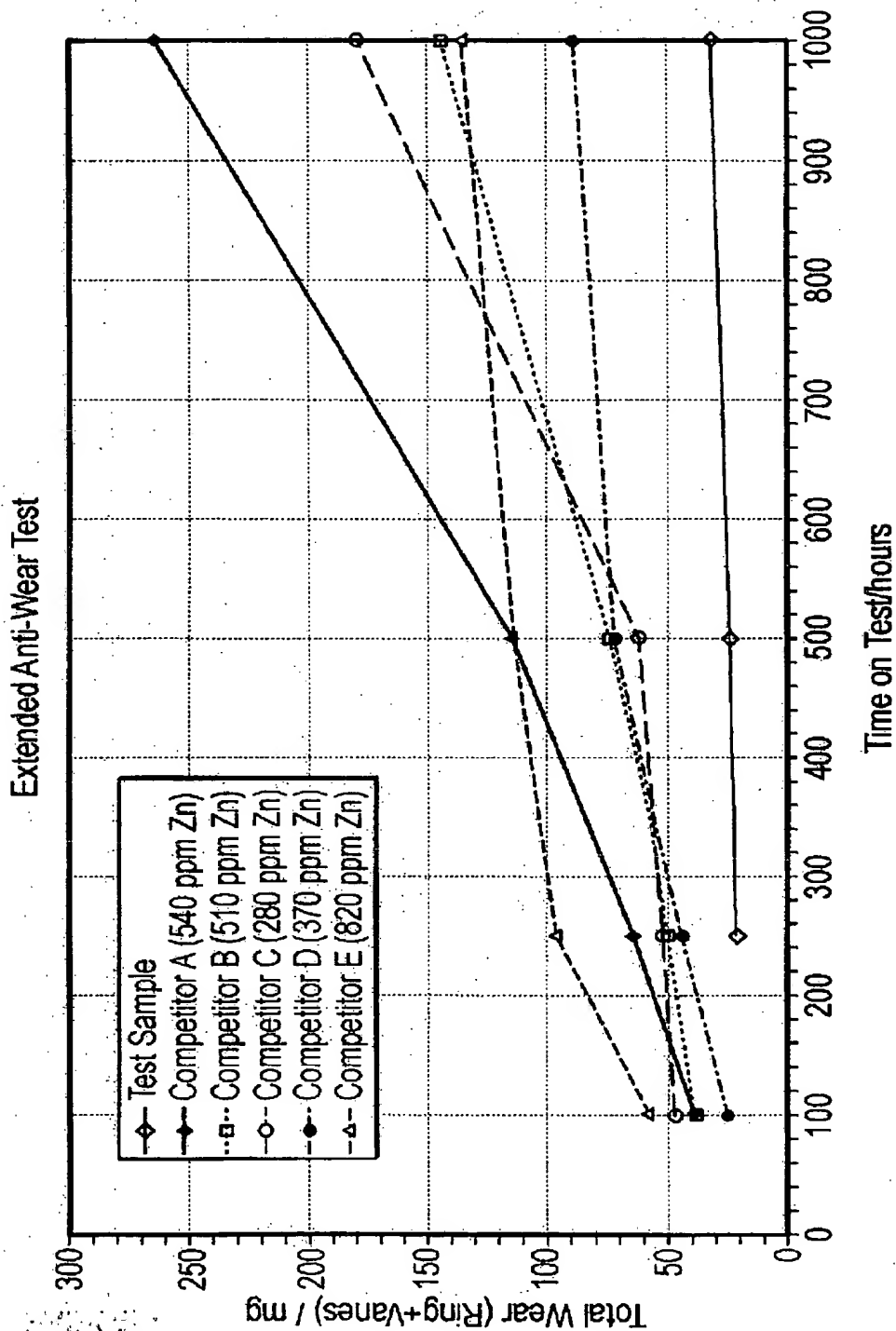


EXHIBIT B

	Composition 1	Composition 2	Composition 3	Composition 4
Magnesium Salicylate	0.06		0.43	
Calcium Salicylate		0.06		
ZnDTP	0.37	0.37		0.43
Anti-rust	0.1	0.1	0.1	0.1
baseoil	balance	balance	balance	balance
Total Wear	15	97	1.9	8.6

Exhibit C